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Study of High Purity Nickel with Various Grain Sizes Using Shear Tests

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Abstract. In this paper, an experimental campaign is described. The high purity Nickel sheet with a thickness of 500 microns is tested at low triaxiality. Thanks to a novel geometry, the axial tensile force is converted to shear the material in a local region. Different series of tests are carried out in function of grain sizes. A strong influence on the global mechanical response can be observed due to different numbers of grains through the thickness of sample. The experimental curves are reproduced by finite element simulations with an isotropic macroscopic Hollomon constitutive law relying on different sets of parameters. Next, a small representative volume is considered. Using the identified parameters for each grain size, a sandwich approach is applied. The results of the single constitutive law approach and the sandwich approach are compared.

INTRODUCTION

The interest of a better understanding of mechanical behavior of metal material at small length scale (typically close to the size of grains inside the material) is growing with the demands of the micro mechanical devices, e.g. in the microelectronics field, in the automotive sector, in the biomechanical applications and so on. The mechanical behavior appearing at the macroscopic length scale of a crystalline aggregate can be regarded as the result of the mechanical behavior of each individual grain at the microscopic length scale. The effects of the grain features, such as the shape and size of grains, the grain orientations, material textures, the distortion of lattice, the densities of grain boundaries and dislocations, on the macroscopic mechanical behavior are quite limited when the material length scale largely overweighs the grain size. On the contrary, the mechanical behavior of each individual grain is more dominant when the grain size is comparable to the material size. Hence, the mechanical behavior of material at micro-/meso length scale depends on the properties of grain aggregate [1, 2], e.g. the number of grain in the material, the orientations of each individual grain, the textures, the slips of lattice and the dislocation densities etc.

The relationship between part size and grain size is usually a key feature to describe the size effect at small length scale. Many efforts have been focused on this topic within the literature. Keller et al. [3] performed micro tensile tests with thin nickel samples with high purity. In this research, the sample thickness is set to 500 microns. Different thickness to grain size ratios are obtained by varying the grain size. The authors reported that with a decrease in number of grains through thickness, a strong decrease in flow stress can be observed. It also modifies, e.g. the work hardening stages, the dislocation structures etc. Chan et al. [4] carried out a series of compression tests by using pure copper cylindrical samples. Their results show that the final shape of cylinders after compression changes from circular section to irregular section with the decrease in section diameter to grain size ratio. Such modifications are mainly due to the inhomogeneous deformation of material in the case of oligo-crystal material. Keller et al. [5] employed a surface

effect model, also called sandwich model, to approach the size effect in deep drawing process. They considered that the overall mechanical behavior can be regarded as the resulting of surface grains and core grains. Due to the escape of dislocations at free surface, the surface grains result in a lower flow stress compared to the one of core grains. The surface grain proportion is determined by the grain size and the sample material size. Differently, Meyers and Ashworth [6] proposed a composite model. They declare that the mechanical behavior of a crystalline aggregate is divided into two parts, viz, a part of continuous grain boundaries and a part of discontinuous interiors of grain. The flow stress of crystalline aggregate is thus the sum of the flow stress from grain boundaries multiplied by the volume fraction of grain boundaries and the flow stress from interior of grains multiplied by the volume fraction of the interior of grains. Jiang et al. [7] has demonstrated that this composite type approach is capable of predicting the influence of the grain size on the yield stress.

In the present study, first of all, a high purity Nickel sheet (99.99 wt% Ni) with a thickness of 500 microns is tested at low triaxiality condition. Thanks to an exotic geometry, the tests are carried out with an axial electromechanical testing machine which is also used for the uniaxial homogeneous tensile test and large tensile test. The interest of using a common experimental equipment for different types of tests is to minimize the machine error impacts. In order to convert the axial loading path to shear the material, a new design of the conventional geometry of the shear test samples has been developed. The constraints, both from the experimental equipment and from the sample dimensions are taken into account during this design. Different geometries are proposed and compared in the work of [8]. Based on the comparisons, an optimal geometry is used hereafter. Then, with an isotropic macroscopic Hollomon constitutive law, the different mechanical behaviors are reproduced by the finite element simulations. In the end, the sandwich (surface effect) approach [5] is employed on a representative volume to predict the size effect.

DESCRIPTION OF EXPERIMENTAL SETUP AND SPECIMEN

The experimental campaign was carried out in the Laboratory for Materials and Structures of University of Liège. As illustrated in FIGURE 1, the shear tests were performed with a uniaxial tensile test machine ZWICK Z600 with a capacity of 600 kN. In order to accurately measure the low force level experiment, the machine is coupled with a cell force of 20 kN. During the test, the samples are deformed with a ramp force having a slope of 5 N/s. The corresponding displacements are measured via the conventional extensometer as shown in FIGURE 1 (b). The initial distance between the two measure arms of the extensometer is set to 25 mm.

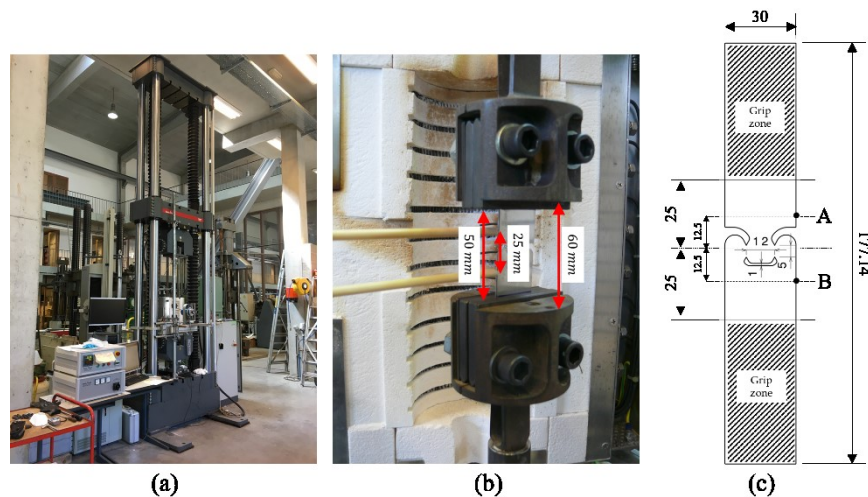


FIGURE 1. (a) Uniaxial tensile test machine ZWICK Z600 (600 kN), (b) the grips and the positions of the measure points of extensometer, (c) the geometry of the shear sample, the dimensions are in mm.

The geometry of the shear sample is presented in FIGURE 1 (c). In order to prevent the sample slipping during the test, the clamping zones are larger than the measured zone. Thanks to the symmetry, there are two shear zones defined in the sample. Each shear zone has a height of 5 mm. The distance between the two shear zones is set to 12 mm to prevent any potential out-of-plane displacement that was observed for the other sizes. The two points A and B indicate the contact positions with the extensometer to measure the elongation of sample.

The samples are annealed in vacuum at different temperatures ranging from 600 °C to 1050 °C. Hence, 5 different grain sizes are obtained between 50 μm and 300 μm .

NUMERICAL MODELING

Description of Finite Elements Model

In this section, the numerical modeling of the experimental test is presented. In this study, the grain boundaries and textures are not taken into consideration, only isotropic macroscopic Von-Mises law with different hardening laws are associated with each grain size. As shown in FIGURE 2 (a), the clamping zones are not taken into account in the finite element simulations. Thanks to the symmetrical geometry, only half width of the sample is modeled. The thickness is meshed by three layers of elements. The shear zone is refined with smaller element size as shown in FIGURE 2 (b). The extensometer measures length between point A and B. The elongation of the sample is also measured by these two points in the numerical simulations.

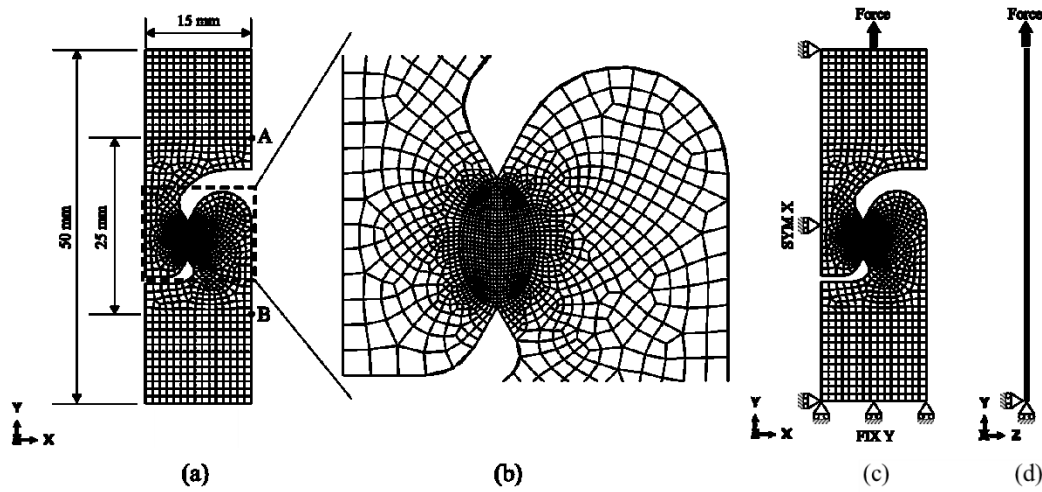


FIGURE 2. (a) Finite element meshes of half sample, (b) zoom for the shear zone, (c) view in X-Y plan of boundary conditions, (d) view in Y-Z plan of boundary conditions.

The boundary conditions are described in FIGURE 2 (c) and (d). The bottom surface is fixed in tensile direction. The symmetrical condition is applied on the left surface along X direction. The sample is deformed by the constant measured force from experiment.

Identification of Material Parameters

TABLE 1. Identified parameters for different grain sizes.

Grain size [μm]	50	70	100	200	300
c [MPa]	755	695.5	678.5	1058	900
n [-]	0.48	0.469	0.471	0.6	0.6

The Hollomon function, defined in equation (1), is used as the isotropic macroscopic elastic-plastic constitutive law.

$$\sigma = c \cdot \epsilon^n \quad (1)$$

In equation (1), σ and ϵ represent the macroscopic stress and strain respectively, c and n are two material parameters. After identifying the parameters for each experiment of different grain sizes, the numerical results can perfectly reproduce the experimental force-displacement curves as shown in FIGURE 3 (a). An example of the shear stress field is illustrated in FIGURE 3 (b) and (c) for the grain size of 50 μm and for a given imposed force of 200 N.

A homogenous distribution of stress field can be observed at the center of the shear zone. The parameters for different grain sizes are summarized in TABLE 1.

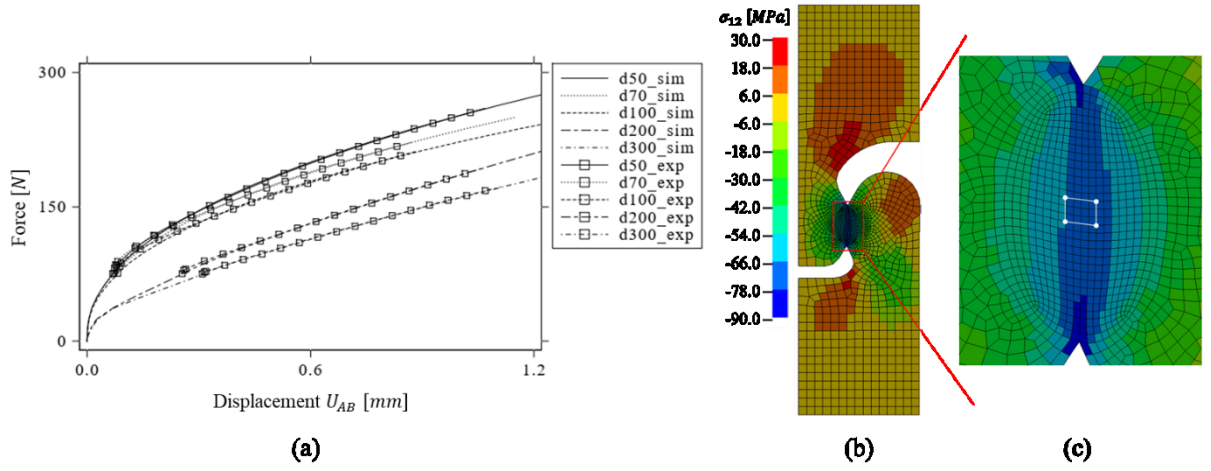


FIGURE 3. (a) Comparison between experimental and numerical results, (b) The shear stress field for 50 μm grain size for a given imposed force of 200 N, (c) the zoom of deformed shear zone in (b).

APPLICATION OF SURFACE EFFECT APPROACH

Since the shear stress field is homogenous at the center of the shear zone, a representative volume is considered in FIGURE 4 (a). The dimensions are set to 0.5 mm in each direction and the Z direction is considered as the thickness direction as in FIGURE 2 (a). The simple shear boundary conditions are imposed on the representative volume. The right surface is fixed in X and Y directions. The back surface is fixed along Z direction in order to prevent the rotation around X axis. A displacement orientated along the positive direction of Y axis is imposed on the left surface to shear the material. For the meshes, in this set of simulations, the representative volume is meshed by 21 elements in the thickness direction (along Z axis) and 14 elements in the two other directions (along X and Y axes). In order to obtain a homogeneous shear stress field in the representative volume, additional boundary conditions had to be imposed numerically. The distortion of the left and front surface are prevented by enforcing a uniform displacement along X direction of the left surface and a uniform displacement along Z direction of the front surface. Except the right surface, the other lateral surfaces are thus forced to deform along Y direction without any rotation.

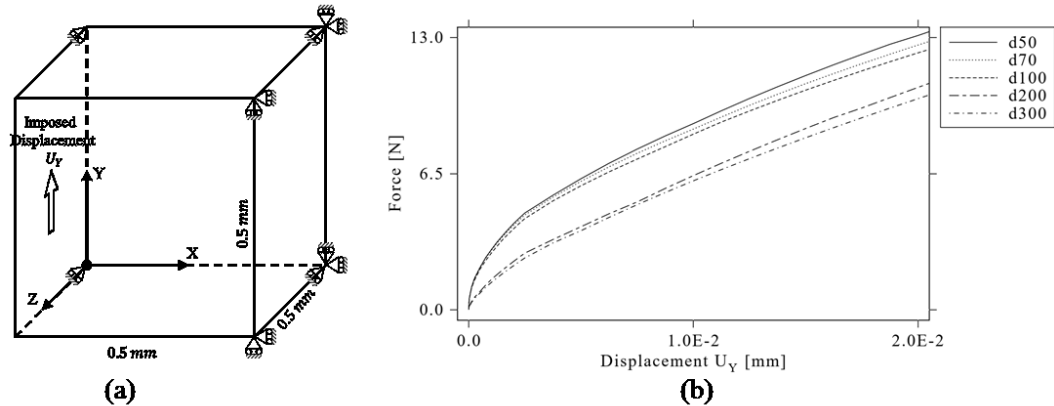


FIGURE 4: (a) Representative volume with dimensions of 0.5 mm in each direction, (b) predicted force-displacement curve by the representative volume for different grain sizes.

The identified material parameters (TABLE 1) for different grain sizes are applied on this representative volume, a measure of the computed global force is drawn in FIGURE 4 (b). FIGURE 3 (a) and FIGURE 4 (b) show the similar variation tendencies.

In the second step, in order to apply the sandwich approach reported in [5], the two extreme cases in FIGURE 4 (b) are considered as the surface grain behavior and the core grain behavior. More precisely, the $300\text{ }\mu\text{m}$ grain size is assumed as the surface grain behavior since the annihilation of dislocations on free surface can usually result in a lower flow stress. On the contrary, the core grains should have a higher stress compared to the surface grains since the grain boundaries act as obstacles for the motion of the dislocations. The accumulation of dislocations at grain boundaries can generate a high back-stress. Therefore, the $50\text{ }\mu\text{m}$ grain size curve is considered as the core grain behavior. The parameters for surface grain layer and core grain layer are listed in TABLE 2.

TABLE 2. Material parameters for core grain layer and surface grain layer.

	c [MPa]	n
Core grain layer	755	0.48
Surface grain layer	900	0.6

Identical representative volume and boundary conditions as in FIGURE 4 (a) are applied. Trials of using the two sets of material parameters to predict the grain size effects by changing the volume fraction of the surface grain and core grain layers were performed for the intermediate cases (grain size of $70\text{ }\mu\text{m}$, $100\text{ }\mu\text{m}$ and $200\text{ }\mu\text{m}$). The representative volume is meshed as shown in FIGURE 5.

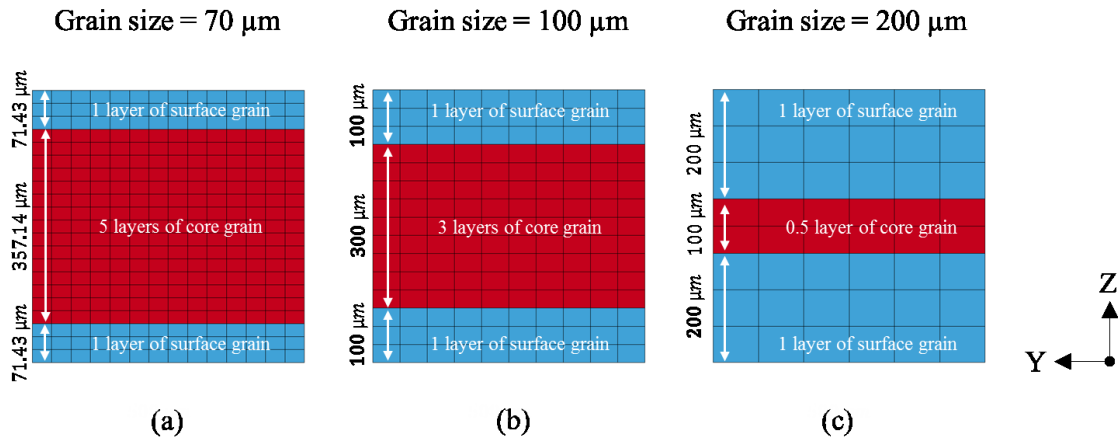


FIGURE 5. Meshes for (a) $70\text{ }\mu\text{m}$ grain size, (b) $100\text{ }\mu\text{m}$ grain size and (c) $200\text{ }\mu\text{m}$ grain size for the surface effect approach.

For the grain size of $70\text{ }\mu\text{m}$, the representative volume is discretized into 7 layers of grains in each direction. For the grain size of $100\text{ }\mu\text{m}$, the volume can be divided into 5 layers of grains in each direction. For the grain size of $200\text{ }\mu\text{m}$, 2 layers of surface grains with a dimension of $200\text{ }\mu\text{m}$ are assumed and the residual volume is considered as the core grain layer. As in [5], each grain is assumed to have a simple parallelepipedic shape. It is meshed with $2 \times 2 \times 3$ elements (along X, Y, and Z axes respectively). The material parameters of the core grain layer are applied in the red volume in FIGURE 5, while the blue volume is modeled by the material parameters of the surface grain layer.

The results obtained by the sandwich model and by the approach with a single law associated to the grain size are compared in FIGURE 6. FIGURE 6 (b) and (c) illustrate the shear stress field without and with the sandwich model respectively. Two different stress values (core and surface) can be distinguished with the sandwich approach while the single law case presents anyway heterogeneous stresses related to different constraints for core and surface material points. However, it is clear in FIGURE 6 (a) that the sandwich approach is unable to accurately predict the grain size effects, even if the global tendency is correct. It may be explained by the following reasons: (i) in [5], this approach is applied to a simple tensile tests where textures evolve less than in the shear state; (ii) Keller's surface law was identified from the 1-grain-through-thickness sample and the core law from the 27-grains-through-thickness case which is far more representative than the current extreme cases of 1.6 and 7 grains. Finally in [5], the sandwich model gave also the surface law only to a fixed thickness depth of the surface grains.

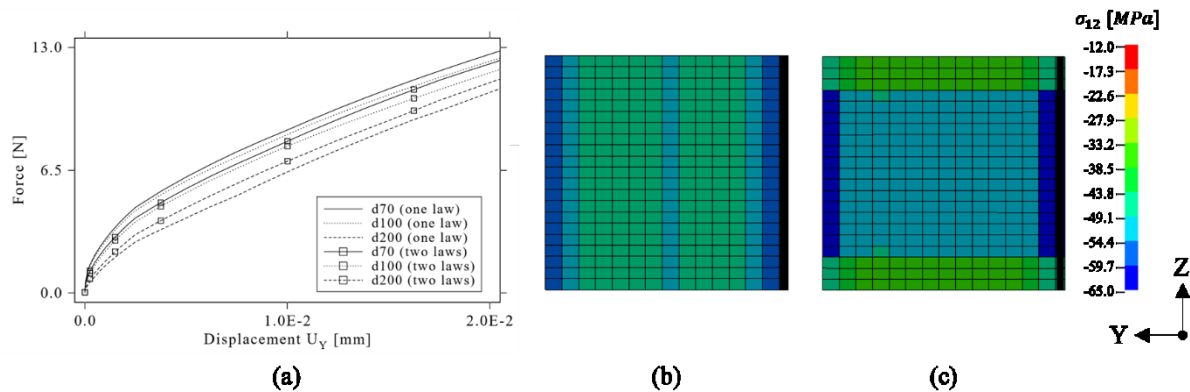


FIGURE 6. (a) Force-displacement curves with and without using surface effect approach, (b) shear stress field for a given imposed displacement of 0.02 mm for the grain size of 70 μm without sandwich approach, (c) idem with the sandwich approach.

CONCLUSION

The shear tests are carried out on a uniaxial tensile test equipment thanks to a novel geometry for the shear sample. Out-of-plane displacement is prevented by the proposed geometry. Different nickel sheets with grain sizes from 50 to 300 μm with high purity are tested. With a constant thickness, the grain size has a strong influence on the mechanical behavior at low triaxiality. Two different sets of experimental force-displacement curves can be observed. A macroscopic constitutive law is identified for each grain size case and the grain size effects are reproduced by finite element simulations. With the identified parameters, a variant of the sandwich approach reported in [5] is checked on a representative volume. The results demonstrate that this too simple approach fails to predict the grain size effect in the present study and it justifies the ongoing research devoted to gradient crystal plasticity model.

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